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Short rotation forestry feedstock: Influence of particle size segregation on biomass properties

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HIGHLIGHTS

- SRF is highly heterogeneous, due to presence of bark and occasionally foliage.
- The younger the SRF is harvested, the more heterogeneous is the feedstock.
- Particle size fractions from ground SRF are affected by the heterogeneity.
- Fine particles below 0.2 mm are particularly well differentiated.
- Removal of fine particles can improve suitability for thermochemical conversion.

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ABSTRACT

Short rotation forestry (SRF) is a promising feedstock for production of biofuels via the thermochemical route. Five poplar biomasses (SRF of different clones and ages, and debarked wood) were ground and separated into three particle size fractions: <0.2 mm; 0.2–0.4 mm; >0.4 mm. The characterization of these samples was performed to evaluate the quality and homogeneity of SRF feedstocks. Some major properties related to thermochemical processes were measured: chemical composition, organic and inorganic elemental compositions. The heterogeneity in SRF feedstock properties, resulting from high bark content, appeared to be transferred to particle size fractions. The results obtained highlighted that fine particles below 0.2 mm had very specific properties, close to those of bark. The removal of this fraction would result in a more homogeneous feedstock, avoiding the issue caused by segregation risk for process stability. Such removal of small particles would also modify the biomass properties by reducing bark amount, improving the suitability of SRF feedstock for thermochemical conversion.

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1. Introduction

Thermochemical processing is a very efficient route for producing bioenergy from lignocellulosic biomass. The direct combustion is currently the most popular process but the interest for other thermochemical processes such as gasification or pyrolysis is growing fast [1]. The production of energy by thermochemical conversion of biomass is increasing worldwide and requires the biomass supply to increase as well. To enable such large scale biomass supply, some efforts are focused on diversification of biomass feedstocks. In this context, the energy crops have a good potential for regular and large scale lignocellulosic biomass production [2]. Among energy crops, short rotation forestry (SRF) could be a promising feedstock for the thermochemical conversion sectors [3–6]. It offers the possibility of a regular supply of biomass that is close to wood in terms of composition: that makes SRF particularly interesting to diversify supply of the current wood-based energy production. Also, SRF would only slightly competes with other sectors such as the food industry or the forestry timber. SRF consists to dense plantations of trees, regularly harvested: 1–5 years rotation for recent bioenergy purposes, and up to 10 years in a more classical growth model like the one used for pulpwood production [7]. Poplar is one of the most common SRF trees in temperate areas like France, due to the fast regrowth of its shoots and its good suitability to cultivation conditions, despite its susceptibility to pests [8,9].

In order to optimize the thermochemical conversion of diversified feedstocks, biomass pretreatment steps must be efficiently designed and well chosen: drying, grinding, pelletization, flash

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pyrolysis and torrefaction [10–13]. The application of these pretreatments has to be adapted to the biomass type and to the technology used for biomass conversion or to the production objectives. Anyway, drying or grinding are essential for most of the process chains. Grinding is an easy step to include in the basic industrial process chain, but it can also have significant impact on biomass properties. Indeed, particle size segregation of the ground biomass can occur during the transportation or conversion steps, thus modifying the properties or creating heterogeneity in the sample [14,15]. Such modifications and heterogeneity of biomass properties may then have a noticeable impact on studies about understanding and modeling of further thermochemical processes. Poplar SRF contains both wood and a significant amount of bark, that makes it a particularly heterogeneous biomass. Wood and bark fractions of SRF poplar are known to have different characteristics and behavior during thermochemical treatment: mass loss kinetics, composition of the products released [16,17]. Particle size segregation would then be a particularly relevant issue concerning the use of SRF feedstocks for thermochemical conversion.

Several biomass properties are known to have an impact on thermochemical processes. Depending on the process considered, it can be organic elemental composition, structural lignocellulosic composition or ash yield and inorganic elemental composition, among others [18–21]. The accurate knowledge of these properties is required for studies that aim to understand and model the impact of biomass properties on process reactions, products yields, etc. If some industrial processes can be more flexible in regard to accuracy of biomass characterization, the control of properties remain critical for many processes. The stability of properties can also be required during the conversion of a feedstock, to ensure constant quality of products. Increasing the flexibility of a process in regards to the range of biomass properties acceptable, or having real-time adaptation of conditions of processing to the biomass properties changes can be quite expensive. An accurate control on biomass properties would also enable advanced applications such as biomass blending to improve biomass suitability to processes.

This study focuses on the control of properties variability within a SRF feedstock. Sources of variability can be classified according to their origin and associated possibilities of control. The natural variability of a biomass feedstock, linked to its growing conditions and to its natural diversity, is an unavoidable processing constraint. For the poplar SRF feedstock considered, natural variability can be illustrated with different poplar clones, grown on different soils and harvested at different ages. Previous studies have shown that the clonal species and age of SRF poplar tend to have a moderate influence on biomass properties and thermochemical treatments [16,22]. An other source of variability – that may be qualified as artificial – comes from the particle size segregation phenomena that can appear during transportation or conversion steps. In this study, the influence of grinding and segregation on SRF poplar physico-chemical properties was evaluated and compared to variability from cultivation conditions (clone, age, soil quality, etc.). This was achieved by dividing ground poplar samples into different particle size fractions which were then characterized. The analyses focused on some physico-chemical properties that are known to impact thermochemical processes: structural lignocellulosic composition, elemental organic composition, ash content and inorganic elemental composition [18–21]. The results will help to determine whether ground SRF poplar can be considered as a feedstock with homogeneous properties or not, thus impacting on further works of characterization, processing and modeling based on this biomass. This work will also provide information on the pertinence and efficiency of the currently used biomass sampling techniques and standards (XP CEN/TS 14780).

2. Materials and methods

2.1. Woody samples

The set of woody biomass samples used for this study was representative of the available French SRF poplar (*Populus* spp.) feedstocks: 4 SRF poplar of different ages and clones completed with poplar debarked wood Table 1. Two-year poplars 2*y-Po*(*a*, *b*, *c*) grown in the same place and older SRF 12*y-Po* grown in another place. All fields had similar latitude (about 49°) and similar alluvial soil. All samples were shredded and naturally dried in the field for some weeks, and then samples weighing some hundreds kg were taken for experiments. The woody chips were then industrially air-dried to 15–20% moisture content (wet basis) before being ground to millimetric particle size (hammer-mill for 1*y-Po*(*a*) and knife-mill for the others). Some kg of ground biomass were sampled at this point for study purposes. Debarked wood sample *Po–W* was obtained from mature poplar chipped and debarked as though for pulp–paper manufacture. The age of poplar was estimated about 30 years, grown in France on alluvial soil. Some kg of chips were sampled and ground (knife-mill) to millimetric size for study purposes.

The poplar samples were ground to obtain a common particle size distribution of sawdust Fig. 1, with average particle size of 1 mm. This type of sawdust is suitable for pellet production or for injection in some gasification reactor technologies. Each sample of ground poplar was then divided into 3 particle size fractions (small/<0.2 mm; medium/0.2–0.4 mm; large/>0.4 mm) by vibrating sieves (70 and 40 mesh sieves were used) with a method derived from standard XP CEN/TS 15149-2. These particle size fractions appeared to be relevant to the study of segregation phenomena, considering the initial particle size distribution of ground biomass. In addition to the set of poplar samples and their particle size fractions, a sample of bark was also characterized. This bark fraction (2*y-Po*(*a*)*-B*) was manually sampled from 2*y-Po*(*a*) SRF; bark mass fraction on a dry basis accounted for about 25% of the global sample weight of 2*y-Po*(*a*) while only about 10–15% for the older SRF 12*y-Po*. Finally, all samples were ground again to reach the particle size required for the different analyses: typically below 1 mm.

2.2. Analytical methods

The following analyses were performed on each particle size fraction, and also on non fractionated poplar samples. Unless otherwise specified, all results were given as mass fractions of dry material. The properties of these non-fractionated samples, termed "raw samples", were compared to those from reconstituted from fractions, to control analytical quality: $P_{rec} = X_{small} \cdot P_{small} + X_{medium} \cdot P_{medium} + X_{large} \cdot P_{large}$ (P_{rec}: reconstituted property, $P_{fraction}$ and $X_{fraction}$: property and weight percentage of small, medium and large particle size fractions).

- Proximate analysis: Moisture content was determined by a method derived from standard NF EN 14774. These data were used to express all results of the further analyses on dry basis.

Table 1
Origin (age and species) of poplar samples. *P.d*: Populus deltoides; *P.n*: Populus nigra; and *P.t*: Populus trichocarpa.

Ref.	Feedstock	Species
2*y-Po*(a)	SRF 2 years	*P.d* × *P.n* (I214)
2*y-Po*(b)	SRF 2 years	*P.d* × *P.n* (Koster)
2*y-Po*(c)	SRF 2 years	*P.d* × *P.n* (Dorskamp)
12*y-Po*	SRF 12 years	*P.t* × *P.d* (Beaupré, Boelare)
Po–W	Wood 30 years	*P.d* × *P.n* (I214)

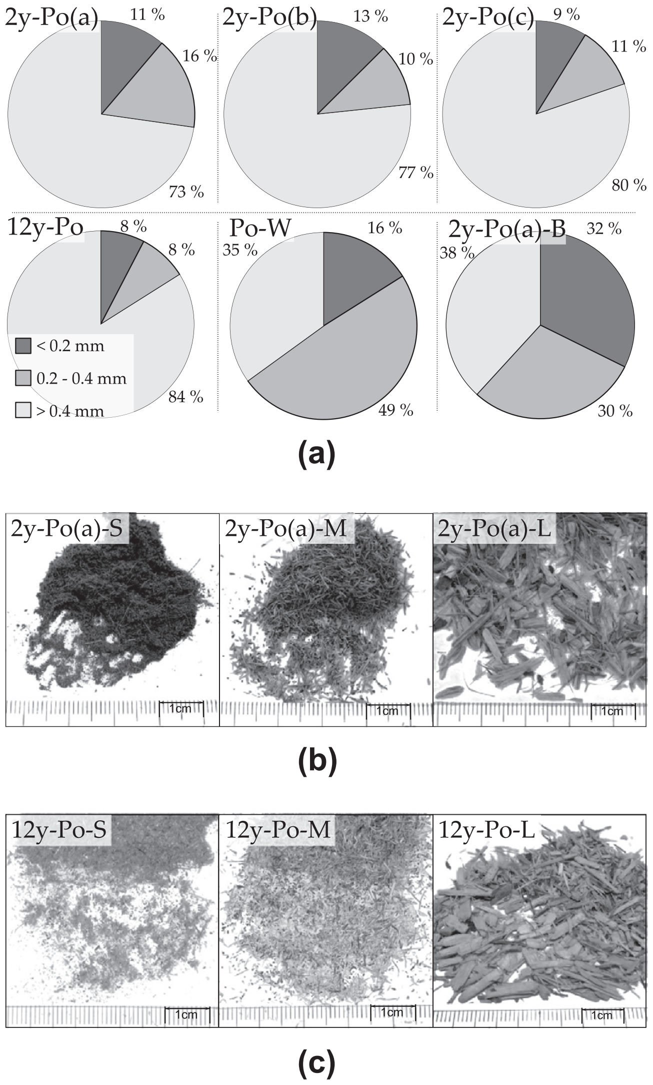

Fig. 1. Ground poplar description: (a) particle size distribution of poplars sawdusts, measured by vibrating sieving; pictures of sawdust particle size fractions (small, medium and large) from (b) 2*y-Po*(*a*) and (c) 12*y-Po* poplars.

Ash yield was determined by combustion of dried non-extracted wood at 550 °C in accordance with standard XP CEN/TS 14775 for solid biofuels.

- Structural lignocellulosic composition: The extractive content was measured using an ASE (Accelerated Solvent Extractor) extraction with 2 cycles at 1500 psi, firstly with water at 110 °C then with acetone at 95 °C. Solvents were evaporated, then the residues were weighted and reported as extractive content. The lignin and polysaccharides contents were measured on the samples previously extracted. The lignin content was determined by weighing solid residue after sulfuric acid dissolution of samples, according to the Klason method described in TAPPI standard T222 om-83. The estimation of cellulose and hemicelluloses contents relied on a method derived from TAPPI standard T249 cm-85: extracted wood samples were totally hydrolyzed in sulfuric acid and sugar monomers were then quantified by chromatography. Cellulose and hemicelluloses contents were estimated by modeling from sugar quantification. Acetyl groups from xylan (main type of hemicelluloses in hardwood) were also quantified by chromatography of the hydrolyzed material and added to the hemicelluloses content. The carbohydrates results were normalized to obtain a dry weight basis fully corresponding to the extractive, ash, lignin and polysaccharides contents. Without normalization, the average mass balance was about 90% (*cellulose* + *hemicelluloses* + *lignin* + *extractive* + *ash*) of the dry material. All analyses of lignocellulosic composition were repeated six times in order to enable statistical analysis of data.
- Elemental analysis: The carbon, hydrogen and nitrogen contents were measured according to standard XP CEN/TS 15104, and the oxygen content was obtained by difference (C + H + O + N + Ash = 100%). The sulfur and chlorine contents were measured by ion chromatography (method derived from standard XP CEN/TS 15289). Quantification of a selection among major inorganic elements – calcium, potassium, magnesium and phosphorus [23] – was achieved using the ICP–AES method after mineralization (method derived from standard XP CEN/TS 15290). The silicon content was analyzed by ICP after alkaline melting and mineralization, to enable quantification of all the silicon in the biomass (including the silicon part that is resistant to the classic mineralization method, [24]). The sulfur, chlorine and inorganic elements analyses were repeated 6 times, to enable the same statistical analysis as for structural lignocellulosic composition. The organic elemental analysis (C, H, and N) were repeated only twice because no significant variability was expected (below experimental uncertainty). Due to the complexity of this analysis, only single measurement was performed for silicon.

2.3. Statistical data processing

Where possible, each analysis of particle size fractions was repeated six times in order to compare measurement uncertainty (from standards of analytical methods) and statistical dispersion. This comparison made it possible to determine whether the sample was significantly heterogeneous or not, compared to a standard homogeneous sample. It was considered important to assess the quality and reliability of the characterization, because SRF samples may be especially heterogeneous. The statistical dispersion of data was represented by two standard deviations (STDs), so that approximately 95% of the measured values would be in the interval. Outliers were detected with help of Dixon's *Q* test at 90% confidence, and removed if necessary.

A student *t*-test was then performed to estimate the probability of a null hypothesis (no significant difference between averages) between the compositions of particle size fractions. This test gave statistical confirmation of apparent differences if $p - value < 0.05$ (null hypothesis rejected with 95% confidence).

3. Results and discussion

3.1. Differentiation of SRF particle size fractions

3.1.1. Structural composition

structural composition Fig. 2 of poplar SRF particle size fractions showed significant differences, especially as regards the cellulose and lignin contents. Cellulose content increased with particle size in fractions from SRF samples (2*y-Po*(*a*, *b*, *c*), 12*y-Po*), ranging from 32% to 48%. For each SRF sample, the variation range between particle size fractions was about 10–13% wide. Small particles were well differentiated from other particle size fractions and there were also significant (according to student *t*-test) differences between larger particles and medium ones.

Lignin was the second largest component in the samples, ranging from 23% to 35%. Its content was higher in small particle fractions from SRF samples (2*y-Po*(*a*, *b*, *c*), 12*y-Po*) than in larger ones. However, the range of variation of lignin content was lower than

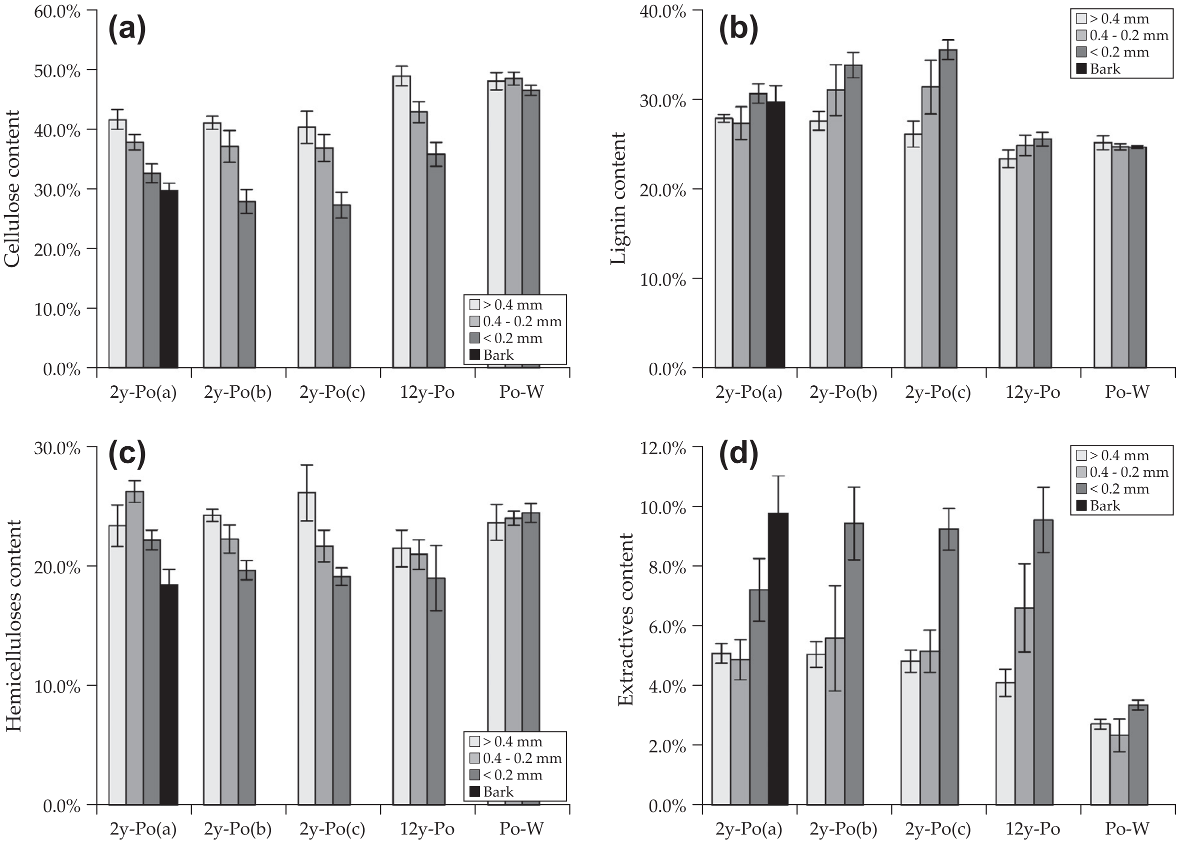

Fig. 2. Structural lignocellulosic composition: (a) cellulose, (b) lignin, (c) hemicelluloses and (d) extractive (water/acetone) contents of particle size fractions. Mass composition on dry basis, plotted with statistical dispersion.

for cellulose content: about 9% between 2*y-Po*(*c*) fractions, but only 2–3% for 2*y-Po*(*a*) and 12*y-Po*. Except between 2*y-Po*(*a*) medium and large particle fractions, all the fractions from SRF samples had significantly (according to student *t*-test) different lignin contents.

The hemicelluloses content of SRF (2*y-Po*(*a*, *b*, *c*), 12*y-Po*) particle size fractions varied over a fairly small range, from 19% to 25 %. The hemicelluloses content of 2*y-Po*(*b*) and 2*y-Po*(*c*) fractions increased with particle size. 2*y-Po*(*a*) and 12*y-Po* fractions did not show clear variations in hemicelluloses content. Some small variations in the composition of the hemicelluloses (xylose, galactose, mannose, arabinose and acetyl contents, not shown) were observed but no generalized behavior appeared. Depending on the sample considered, the results indicated that the xylan (*xylan* = *xylose* + *acetyl*; which had an average proportion in the hemicelluloses of 81%) content may slightly increase with particle size (an increase of +10% of the hemicelluloses proportion in average), to the detriment of other hemicelluloses (mainly galactoglucomannan).

Significant differences were observed on the extractive content of the particle size fractions. All the SRF samples (2*y-Po*(*a*, *b*, *c*), 12*y-Po*) showed the same tendency: small particles had higher extractive content than larger ones (+2 to 5%). However, for young SRF (2*y-Po*(*a*, *b*, *c*)) the extractive content was rather the same for large and medium particles (about 5%).

3.1.2. Elemental organic composition

Some small differences of carbon content Fig. 3 could be observed between fractions of young SRF samples: about 48% in small particles of SRF versus about 50% in large particles. The older SRF 12*y-Po* particle size did not show significant differences in carbon content in small particle size fraction. When calculated on a *dry ash-free basis*, all SRF showed similar trend with about 1% more carbon in small particle size fractions compared to large particle size fraction and even +6% for 12*y-Po* fractions. The hydrogen content in small particle size fractions of young SRF (about 5.5%) was significantly lower than in large particle size fractions (about 6.0%). No significant difference could be observed between particle size fractions of older SRF (about 6.1%). When calculated on a *dry ash-free basis*, no significant difference in hydrogen content could be observed between particle size fractions of any SRF sample. The oxygen content of small particle size fractions of SRF were significantly lower (about 35–37%) than in large particle fractions (about 41% up to 44% in 12*y-Po*). These differences were lower but still significant (particularly in 12*y-Po*) when calculated on a *dry ash-free basis*. So, the differences that could be observed in CHO composition on a *dry basis* were mainly due to the differences in ash yield Fig. 4. Concerning composition of organic matter, a moderate (to large in 12*y-Po*) carbon content increase in small particles was observed, to the detriment of oxygen content.

The nitrogen content was higher in small particles (average 2%) than in large particles (average 0.5%) of young SRF 2*y-Po*(*a*, *b*, *c*). The older SRF 12*y-Po* had lower nitrogen content (0.2%) and no differences could be found between its particle size fractions.

The sulfur and chlorine contents (not shown) were close to their detection limit, and the standard deviation of measurements was much higher than experimental uncertainty. As a consequence and in spite of the number of repetitions performed, no statistically significant difference could be found between particle size fractions. The information useful in biomass characterization was that the sulfur and chlorine contents were very low in poplar.

3.1.3. Ash yield and inorganic elemental composition

In all the SRF samples (2*y-Po*(*a*, *b*, *c*), 12*y-Po*), ash yield Fig. 4 strongly increased while particle size decreased (+5.3 to 7.9%). A

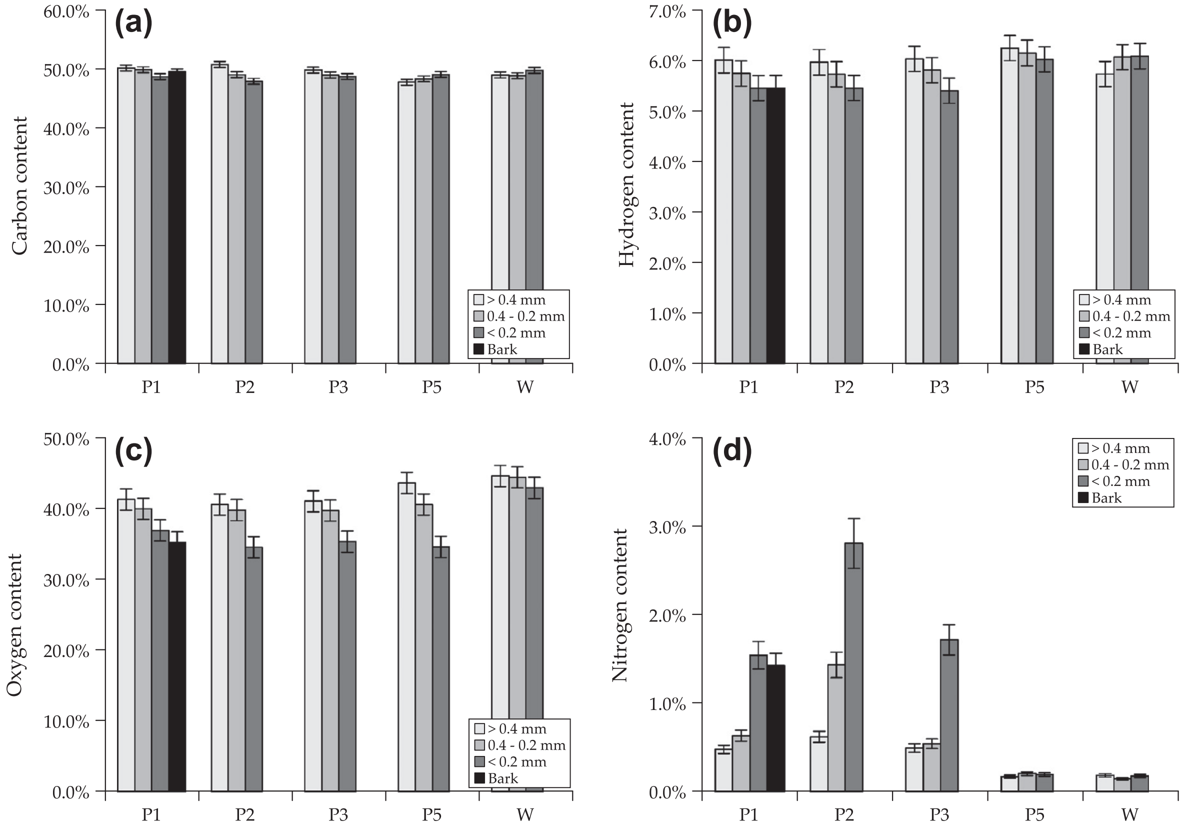

Fig. 3. Organic elemental composition on dry basis: (a) carbon, (b) hydrogen, (c) oxygen (by difference), and (d) nitrogen contents in particle size fractions. Mass composition on dry basis, plotted with experimental uncertainty of measurements.

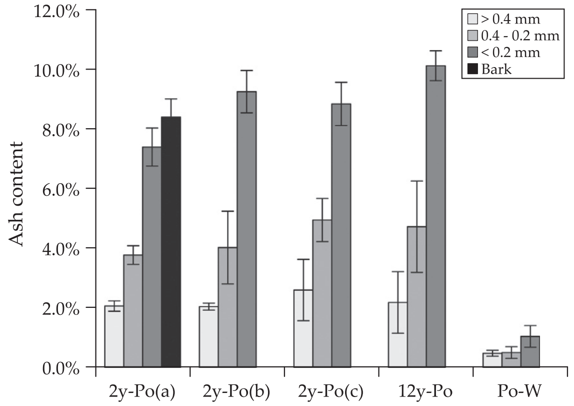

Fig. 4. Ash yield (550 °C) of particle size fractions. Mass composition on dry basis, plotted with statistical dispersion.

similar occurrence was observed with *Po–W*, but with a much lower difference between particle size fractions: the ash yield of the small particles was 0.5% higher than in the two other fractions, which were very similar.

The inorganic elemental analysis of poplar particle size fractions Fig. 5 showed many significant differences in composition between fractions and between samples. Calcium and potassium were the major elements in all samples (respectively 0.15–3.02% and 0.13–0.77%), followed by magnesium and phosphorous (respectively 0.02–0.17% and 0.01–0.33%), as expected [25]. Similarly to the ash yield of SRF (2*y-Po*(*a*, *b*, *c*), 12*y-Po*) fractions, the inorganic elements contents in SRF biomass were generally higher in the small particles than in the larger ones. Silicon appeared to be mainly present in small particles while its content in medium and large fractions was particularly low. This element might be especially impacted by grinding and particle size segregation.

In addition to the elemental composition of the biomass Fig. 5, the main inorganic oxides (CaO and K_2O) contents in ash were calculated Fig. 6. While ash yield and thus inorganic elements contents showed large variations, the oxide composition of ash showed few significant differences between particle size fractions.The K_2O percentage in ashes appeared to be higher in large particle size fractions than in small particle size fractions for all samples. No significant difference in CaO percentage between ash of particle size fractions was observed.

3.2. Impact of age, clone, soil

Few relevant differences in structural lignocellulosic composition could be observed between the 2-year clones Fig. 2. Due to a different initial grinding (hammer-mill versus knife-mill), most of the differences observed between the 2*y-Po*(*a*) clone (differentiation of small particle size fraction on lignin, hemicelluloses and extractives contents less obvious) and the two other 2-year clones could not be attributed to a clone effect. The moderate differences observed in nitrogen content Fig. 3 of the 2-year samples (mainly on small particle size fractions) seemed to be related to a clone effect. The variations in carbon and oxygen content observed between 12*y-Po* particle size fractions were particularly large compared to variations in 2-year samples. It was not possible to determine definitely whether this was due to a clone or age effect. In the context of this study, the clone factor appeared to have limited impact on poplar SRF properties.

Some differences between poplar samples could be attributed to the age of trees. Both 12*y-Po* and *Po–W* had significantly higher

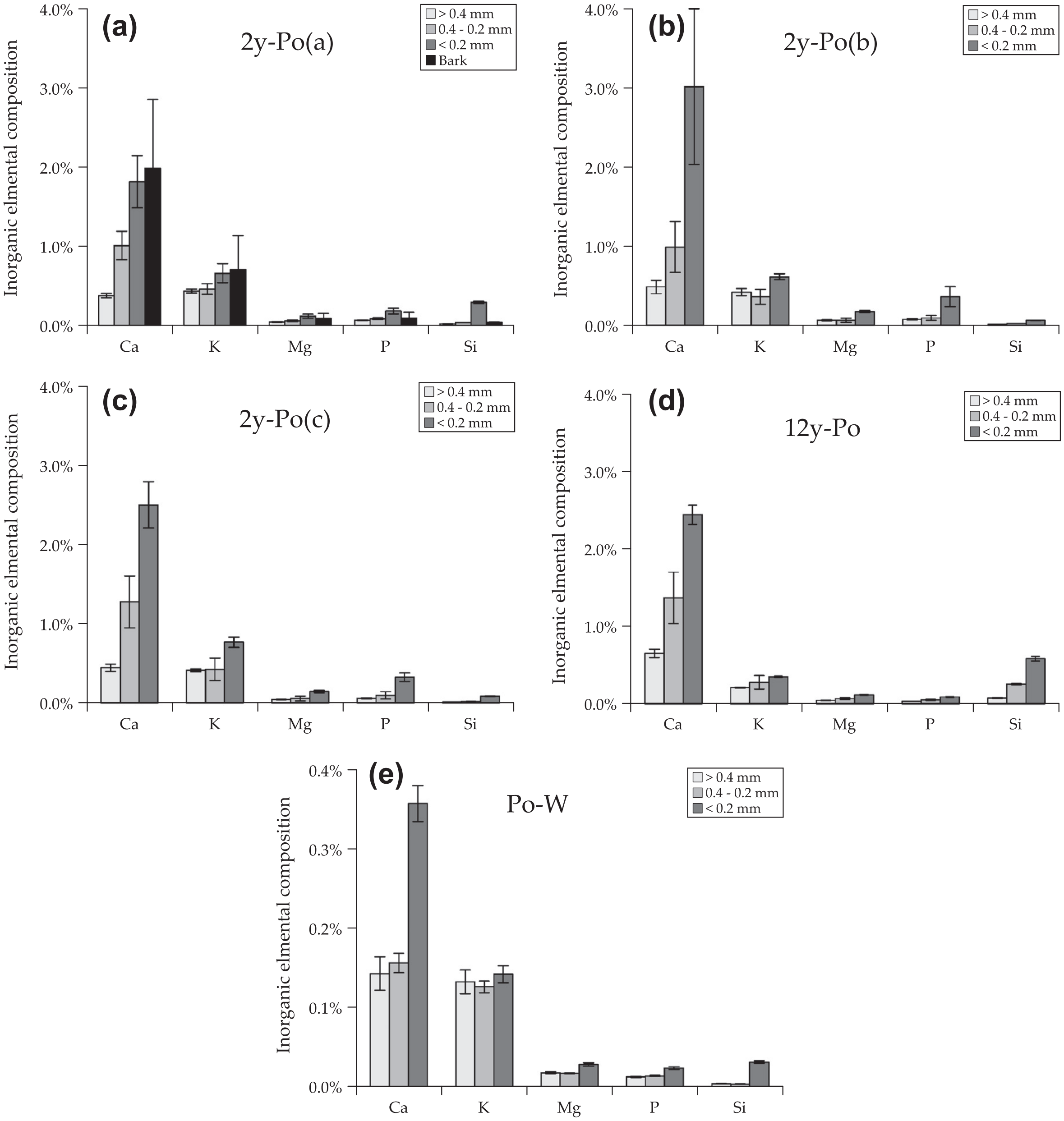

Fig. 5. Inorganic elemental composition on dry basis: Ca, K, Mg, P and Si. (a) SRF 2*y*-*Po*(*a*) particle size fractions and bark, (b) SRF 2*y*-*Po*(*b*) particle size fractions, (c) SRF 2*y*-*Po*(*c*) particle size fractions, (d) SRF 12*y*-*Po* particle size fractions and (e) debarked wood *Po*–*W* particle size fractions. Mass composition on dry basis, plotted with statistical dispersion (except Si, single measurement with 5% *rel.* uncertainty).

cellulose content (48%) and lower lignin content (23–25%) than 2-year samples. This could be representative of a maturation of wood and possibly the development of heartwood [26]. The nitrogen content in 12*y*-*Po* and *Po*–*W* was especially low (about 0.2%). Furthermore, *Po*–*W* also had much lower ash and extractives contents and higher hemicelluloses content. This may be explained by the debarked characteristic of this sample since bark is known to have especially high ash and extractives content [16,17]. Similar explanation would be relevant for differences in potassium content that was quite low in 12*y*-*Po* (about 0.2%) and *Po*–*W* (about 0.1%) compared to 2-year samples (0.4–0.7%). Some minor differences in magnesium and phosphorous contents were observed between samples but could not be correlated to age.

Nutrients (N, K, Ca, Mg, P) contents are known to be impacted by multiple factors such as soil quality, irrigation, clone and age [27]. The differences observed in nitrogen and potassium contents were mainly attributed to age, but other factors may also contribute. The differences observed in magnesium and phosphorous contents could occur because of a combination of several factors. Finally, silicon was concentrated in small particle fractions and its content was quite different between samples: 0.01–0.02% in 2*y*-*Po*(*b*, *c*) and *Po*–*W*, 0.05% in 2*y*-*Po*(*a*) and over 0.1% in 12*y*-*Po*. Soil composition and contamination of bark with soil particles would be the most relevant explanation for these differences.

3.3. Role of bark

Unlike the SRF fractions, there were no significant differences of lignocellulosic structural composition between debarked wood *Po*–*W* fractions. Nor was elemental organic composition of this poplar sample affected by particle size segregation. Most of the properties of *Po*–*W* were close to properties of large particles of SRF and very

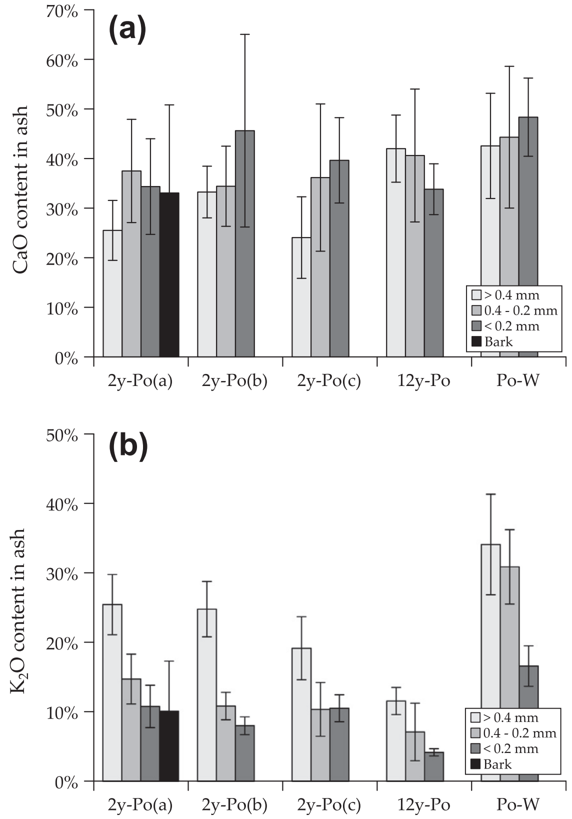

Fig. 6. Ash composition in oxides: (a) CaO and (b) K_2O. Mass composition on ash basis, plotted with statistical dispersion.

different from properties of small particles. For some properties such as cellulose, extractive, ash or nitrogen contents, *Po–W* properties were significantly different to those of large particles from SRF. This could correspond to a difference of maturity of wood: cellulose content would increase with maturity of wood while extractive, ash and nitrogen contents decreased. Thus it appeared that large particles of SRF were very similar to debarked wood.

As mentioned previously, the bark has very different properties from wood, resulting in feedstock heterogeneity in case of feedstock with high bark content [16,17,25]. Indeed, properties of 2*y-Po*(*a*)*-B* were always (except for Si) the most different from properties of *Po–W*, for example 17% less cellulose, 9% more lignin, 5% less hemicelluloses, 7% more extractives, 8% more ash, 1% more nitrogen.

Similarly to the correspondence between debarked wood and large particles from SRF, it was possible to compare bark properties to those of small particles from SRF. Cellulose, lignin, ash and all elemental contents – except silicon – of 2*y-Po*(*a*)*-B* could not be differentiated from properties of small particle fraction of 2*y-Po*(*a*). Small particles from SRF had high extractive contents (from 7.2% to 9.5%), almost as high as extractive content in bark. Results suggested that hemicelluloses content in small particles of SRF could be lower than in large particles. This tendency corresponded to the differences in hemicelluloses content observed between bark (2*y-Po*(*a*)) and debarked wood (*Po–W*).

These observations give evidence that differences in grinding behavior between bark and wood could be responsible for most of the differences in properties of particle size fractions of poplar SRF. Indeed, bark is known to have lower stress resistance than wood, which increases its grindability (this effect can be used to reduce bark content in wood chips feedstocks [28]). Consequently, it could be that the small particle fractions from poplar SRF contain high proportions of bark, resulting significant differences in the properties of this fraction compared to the rest of the SRF feedstock. In accordance with this observation, the grinding of debarked wood (*Po–W*) did not introduce such major properties variations related to particle size.

However, some properties such as ash yield were slightly affected by grinding and particle size segregation of *Po–W*. A similar observation was made in respect of particle size fractions from grassy biomasses [14] which are less heterogeneous than SRF biomass (containing both wood and bark). Consequently, heterogeneity between particle size fractions of SRF would be mainly due to bark specific grindability but grinding of wood could also have a direct impact on properties of particles. Indeed, in contrast to other properties, silicon content variations in particle size fractions did not appear to be driven by bark content. Indeed, silicon content was especially high in small particles but was very low in bark Fig. 5. Subsequently, grinding of poplar (even debarked) appeared to have a direct impact on silicon content. A similar observation could be done, to a lesser extent, for calcium content that was significantly higher in small particles of debarked wood (*Po–W*).

3.4. Specific observations for the use of SRF feedstock

In this study, the impact of external factors (age, clone, soil) on biomass properties appeared much lower than the artificial variability in properties induced by separation of particle size fractions of poplar SRF. However, this artificial variability was similar or even lower than differences in properties that can be observed between woody species and other kinds of lignocellulosic biomasses [29,30]. Most of the raw biomass properties measured were in good agreement with properties calculated from particle size fractions, confirming the reliability of results obtained. However, for several properties analyzed with multiple replicates, statistical dispersion (2σ) of data was regularly higher than uncertainty from standards of analytical methods. Thus, even when sieved into potentially more homogeneous particle size fractions, SRF samples still appeared to be heterogeneous from analytical point of view.

The artificial variability and sample heterogeneity observed between particle size fractions of SRF may have an impact on the accuracy of characterization studies and further studies relying on such characterization data. Indeed, lignocellulosic composition is known to have an impact on torrefaction and pyrolysis processes, while elemental composition of organic and inorganic matter has an impact on gasification processes (thermochemical equilibrium modeling, ash melting and slagging behavior) [18,20,31–35]. In regard to some thermochemical processes, bark content has a negative impact on biomass quality (high ash yield, lower heating value, etc. [16,17]) and its presence should then be minimized.

The grinding behavior of bark and wood is different: bark is more easily ground into small particles, resulting in a rather higher bark content in small particle size fraction [28]. Assuming that bark content was the main source of differences in properties between particle size fractions of SRF poplar and that all these fractions were a combination of bark and wood, a rough correlation of the properties showing significant variability (cellulose, lignin, extractives and ash) gave quite interesting results. According to the cellulose and ash yields, small particle fraction of 2*y-Po*(*a*) (SRF, clone I214) would contain about 63% of bark and 37% of *Po–W* (debarked mature wood, clone I214). Lignin and extractive based correlations gave a bark percentage of around 80–90% in this 2*y-Po*(*a*) small particle size fraction. The same method gave a bark percentage of between 30% and 55% in 2*y-Po*(*a*) medium particle size fraction, and between 20% and 35% for large particle fraction.This was in accordance with observations on particle size distribution and pictures of particle size fractions (Fig. 1 where small particles, rich in bark, appear darker).

Removing small particles below 200 μm from ground poplar SRF feedstock would be equivalent to remove a significant proportion of bark from SRF feedstock: about 6–10% of the initial 10–25% bark content, according to previous estimations. This would avoid process instabilities, due to differences in thermal behavior between bark and wood [16], resulting from the risk of segregation during process. Moreover, the properties of the raw SRF samples were closer to large or medium particles properties than to small particles properties. Thus, the removal of small particles would have significant positive impact on global properties: the feedstock properties would be closer to those of debarked wood chips. A partial removal of the small particle size fraction may help to control the gasification process trough inorganic elements contents that can have catalytic impact on gasification reactions, and ash composition that impact its melting properties, among others. The small particle size fraction, with high ash and extractives contents, may be considered as a co-product of process and used for other applications such as return to soil or green chemistry.

4. Conclusion

The heterogeneity of SRF feedstock resulting from its high bark content appeared to be transmitted to particle size fractions through grinding. Indeed, properties (structural lignocellulosic composition, elemental organic and inorganic composition) of small particles obtained after grinding of SRF were very similar to those of bark. As a result, this kind of feedstock would be highly susceptible to particle size segregation phenomena. Such segregation would impact any analytical study on SRF feedstock, for characterization or thermochemical conversion tests. Stability of thermochemical conversion processes may also be compromised in case of segregation.

The variability of properties between particle size fractions from SRF feedstock was comparable to differences existing between various woody feedstocks but larger than differences between poplar samples studied. Thus, if accurate knowledge of properties is required for a process, such as biomass blending, mixing several poplar feedstocks could be a smaller issue than particle size segregation.

The particles below 0.2 mm were well differentiated from the other particle size fractions and from the global SRF sample: they had higher lignin, extractives, ash, nitrogen and inorganic elements (K, Ca, Mg, P, and Si) contents and lower cellulose, hemicelluloses, carbon, hydrogen and oxygen contents. Their removal prior to the thermochemical conversion would increase homogeneity of feedstock. Bark is often considered as a fuel with lower quality than debarked wood, as regards the thermochemical processes. As the small particle fraction appeared to contain especially high amount of bark, the removal of this fraction would increase the biomass quality and SRF feedstock suitability for thermochemical conversion. The small particle size fraction separated would have high ash and extractives contents that makes it especially suitable for alternative use such as return to soil to compensate mineral export or green chemistry.

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.fuel.2013.04.043.

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